Composition design of Ni–nano-Al2O3–PTFE coatings and their tribological characteristics

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Abstract

The Ni–nano-Al2O3–PTFE composite coatings were prepared by the electrophoretic-electrochemical deposition method. A uniform film of the nano-Al2O3 and PTFE particles was first electrophoretically co-deposited on the substrate of copper. Then, nickel was electro-deposited on the substrate covering with the film of these particles. Using the electrophoretic-electrochemical deposition method can remarkably increase the particle content in the composite coatings. Under certain conditions, the contents of Al2O3 and PTFE particles in the Ni–nano-Al2O3–PTFE composite coating can reach 26.57 wt.% and 13.79 wt.%, respectively. The results of friction and wear tests showed that the Ni–PTFE composite coating exhibited the lowest friction and the Ni–nano-Al2O3 composite coating possessed the best wear resistance. By optimizing the content ratio of nano-Al2O3 and PTFE particles, the composite coating of Ni–nano-Al2O3–PTFE displays preferable low friction and anti-wear behavior.

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1. Introduction

The composite coating is a layer deposited on the surface, in which one or more types of solid particles are uniformly distributed into a metallic or non-metallic matrix [1]. Therefore the performance of the coating can take advantages from both the base material and composites. For example, PTFE or MoS2 particles are often co-deposited with metals to obtain a self-lubricating property [2]. Particles with high hardness, such as diamond, Al2O3 and SiC, have been added to the metal matrix to improve the wear resistance of coatings [3–5]. Of course, the characteristics of the composite coatings depend in part on the properties of the solid particles. Ni–PTFE composite coating can significantly reduce the friction coefficient, while in heavy-load conditions, its wear resistance is poor [6]. The Ni–nano-Al2O3 composite coating shows good wear resistance, but the anti-friction performance expresses no obvious improvement [7]. Therefore, attention has been paid to the composite coatings, which contained both soft and hard particles to acquire friction reduction and anti-wear property as well, e.g., Ni–PTFE–SiC has been used for many years as a high performance coating on automotive engine cylinder liners [8].

Recently, the principle of electrochemical deposition of hard, wear resistant and soft, self-lubricating inclusions in composite metal coating is well established. Lee investigated the co-deposition behavior of dia-
prone to decomposition. Moreover, the composite coatings usually have a high stress [6]. Moreover, the particle contents in the composite coating by using electroless composite plating cannot reach a high concentration, which would seriously influence the performance of the composite coatings.

The electrophoretic–electrochemical deposition method was a two-step process. A film of particles was first electrophoretically deposited on substrate and metal was then electrodeposited in the second step onto the substrate covering the film of particles. Shrestha [18] firstly introduced the method to prepare the Ni based composite coatings, which contain up to 60 vol.% Al$_2$O$_3$ or c-BN ceramic particles. Tian [7] confirmed the validity and reliability of the process. Recently, adopting this process, Ni–diamond composite coating with 46.06 wt.% of diamond particles was prepared by Huang et al. [3] and the coating showed high wear resistance and strong abrasive ability to the opposite contact part.

Compared with the traditional composite electroplating, the two-step method has been proved to be an effective technique for the preparation of composite coatings with high particle content, though the process and facilities of the two-step method are more complex. Since the electrical force is utilized to deposit the particles, the surface properties of particle are sensitive to the electrical behavior. The question is can the two-step method be applied to the soft particles, such as PTFE, and whether it is feasible to prepare multi-composite coatings by using this two-step method. There is little knowledge about it. In this paper, we attempted to prepare the composite coatings of Ni–nano-Al$_2$O$_3$–PTFE by the electrophoretic–electrochemical deposition method. And the hard bearing phase of Al$_2$O$_3$ and soft self-lubrication phase of PTFE were chosen as composite particles. The purposes were to improve the particle contents in the composite coating and to obtain a comprehensive layer with low friction and good wear resistance.

2. Experimental design and procedures

The electrophoretic deposition and electro-deposition processes are used to prepare Ni–nano-Al$_2$O$_3$–PTFE composite coating. First, PTFE and nano-Al$_2$O$_3$ with the particle sizes of about 5 μm and 20 nm were electrophoretically co-deposited on a copper substrate. Then nickel was electrodeposited on the substrate, covering the electrophoretic deposited layer.

2.1. Electrophoretic deposition

During the electrophoretic deposition process, anhydrous ethanol and MgCl$_2$·6H$_2$O were chosen as the dispersing media and the dielectric medium, respectively. Compared with the water-based electrophoretic deposition, using anhydrous ethanol has important advantages since it shows good chemical stability, and low electrolytic gas evolution [19].

Generally, two-component electrophoretic deposition could be realized by two ways, sequential electrophoretic deposition and electrophoretic co-deposition. Sequential electrophoretic deposition means to deposit one type of particles by another. Electrophoretic co-deposition is that two types of particles are electrophoretically deposited at the same time. The sequential deposited film shows that the particle distribution is heterogeneous. The reason could come from the firstly deposited particles, which cause the partial insulation on the substrate. This problem can be avoided by the electrophoretic co-deposition process. Therefore, the electrophoretic co-deposition method was used in the following experiments.

Several issues should be considered for the electrophoretic co-deposition of two types of particles. 1) The electrically charged identity of the particles. The particles charged positively would deposit on the cathode, otherwise on the anode. Therefore, the two types of the particles should have the same electrically charged identity in the electrophoretic liquid. 2) The electrophoretic deposition rate of the particles. The deposition rates of the particles are different due to the different properties. 3) The particle concentration in the electrophoretic liquid. The particle concentrations would affect the final particle content in the layer.

Fortunately, the electrically charged identities of the nano-Al$_2$O$_3$ and PTFE are both cathodical, which makes it possible to form the Al$_2$O$_3$–PTFE composite electrophoretic coating by using the co-deposition method. Fig. 1 gives the schematic illustration of the electrophoretic co-deposition process of nano-Al$_2$O$_3$ and PTFE particles.

The electrophoretic deposition rates of nano-Al$_2$O$_3$ and PTFE were evaluated separately with the same concentration of nano-Al$_2$O$_3$ and PTFE as 2, 4, 6, 8, 10 and 12 g·L$^{-1}$, under the condition of 100 V electrophoretic voltage, 2 min electrophoretic time, and 0.56 g·L$^{-1}$ MgCl$_2$·6H$_2$O concentration. The electrophoretic deposition rate is likely linear to the concentration of the two types of particles and the

![Fig. 1. Schematic illustration of the electrophoretic co-deposition process of nano-Al$_2$O$_3$ and PTFE particles.](Image)
electrophoretic deposition rate of nano-Al$_2$O$_3$ particles is faster than that of PTFE particles at the same conditions (see Fig. 2).

Considering the different electrophoretic rates between nano-Al$_2$O$_3$ and PTFE, the concentration of nano-Al$_2$O$_3$ and PTFE in the electrophoretic suspension was set as 1:6, 1:3, 1:2, and 2:3.

Fig. 3 gives the micro-morphologies of the electrophoretic deposition layers conducted at 100 V electrophoretic voltage, 2 min electrophoretic time, and 0.56 g·L$^{-1}$ MgCl$_2$·6H$_2$O concentration. Fig. 3a shows the single electrophoretic deposition layer of nano-Al$_2$O$_3$, under the concentration of 8 g·L$^{-1}$ Al$_2$O$_3$. It can be found that Al$_2$O$_3$ particles are distributed as the small round ball in the electrophoretic deposition layer. Fig. 3b shows the PTFE electrophoretic deposition layer, under the concentration of 12 g·L$^{-1}$ PTFE. Fig. 3c expresses the co-deposited layer of nano-Al$_2$O$_3$ and PTFE with 12 g·L$^{-1}$ PTFE and 8 g·L$^{-1}$ Al$_2$O$_3$, respectively.

2.2. Electro-deposition

After electrophoretic deposition, owing to the weak binding force between the particles and the substrate, the substrate covered with electrophoretic deposition layer should be put in the plating solution as soon as possible.

The anode and cathode were nickel plate and copper substrate covered with the electrophoretic deposition layer, respectively, as shown in Fig. 4. Because the particles deposited on the substrate are vulnerable to electric field disturbance and easy to drop off the substrate, the anode and cathode are placed horizontally in an evaporating dish. A Watt-type bath was adopted as the plating solution and its main components were shown in Table 1. Array 3645A DC stabilized power was employed as the power source. Cathodic current density for electrodeposition was 1.25 A·dm$^{-2}$. The solution temperature was 40 °C and electroplating time was 2 h.

For the purposes of discussion, the composite coatings prepared under various conditions were numbered as shown in Table 2. Sample No.1 was the Ni–PTFE composite coating, samples No.2–5 were Ni–nano-Al$_2$O$_3$–PTFE composite coatings and sample No.6 was Ni–nano-Al$_2$O$_3$ composite coating.

2.3. Analysis of the coatings

After electrophoretic and electro-deposition, the coatings were ultrasonic cleaned in acetone for 10 min. The surface morphology of the coatings was examined by scanning electron microscopy (SEM) and the particle content was analyzed by an energy dispersive spectrometer (EDS).

Friction and wear tests of the composite coatings were carried out using a ball-on-disk wear tester under dry sliding conditions. A GCr15
steel ball with a diameter of 4.7 mm and Ra 0.025 was adopted as the upper specimen to slide against the composite coatings. The friction tests were performed under a load of 1.88 N at the speed of 94 mm·s\(^{-1}\) for 30 min. The wear tests were performed under various loads of 4.7, 6.6 and 8.9 N at the speed of 105 mm·s\(^{-1}\) for 100 min. The width and depth of wear scars on the coatings were measured using a surface profilometer (Nano-map 500Ls).

3. Results and discussion

3.1. Surface morphology and particle concentration

Fig. 5 shows the surface morphology of the composite coatings prepared by the electrophoretic and electro-deposition under various conditions. The particle contents in different composite coatings are summarized in Table 3. Fig. 5a shows the image of Ni–PTFE composite coating and PTFE particles, in the form of flakes, are embedded in the Ni matrix. The PTFE particle content in the composite coating is up to 31.17 wt.%. Fig. 5b–d presents the surface images of the Ni–nano-Al\(_2\)O\(_3\)–PTFE. It reveals that with the increase of Al\(_2\)O\(_3\) concentration in the electrophoretic solution, the final Al\(_2\)O\(_3\) particle content in the coatings rises from 8.11 wt.% to 13.79 wt.%. Meanwhile, the PTFE particles in the composite coatings maintain about 25 wt.% which decreases a little compared with the Ni–PTFE coating. Fig. 5e shows the surface image of the Ni–nano-Al\(_2\)O\(_3\) composite coating. A large quantity of Al\(_2\)O\(_3\) particles existing in the form of the sphere are embedded in the Ni matrix. The particle content in the coating reaches as high as 44.27 wt.%.

As a summary, the results indicate that the electrophoretic and electro-deposition process could increase the content of particles in the coating layer significantly. In addition, the content ratio of different types in the layer could be adjusted by changing the concentration ratio of the particles in the electrophoretic suspension.

3.2. Tribological performances

It is well known that the combination of different materials could produce the improved mechanical properties, including tribological property. The co-deposition of Al\(_2\)O\(_3\) particles in the deposit greatly improves the hardness and the wear resistance of composite coatings [20]. Polymers especially polytetrafluoroethylene (PTFE), with their non-stick properties are used mainly for dry lubrication [14]. For bearing applications, the coating selected must possess high ultimate tensile strength, hardness and low dry coefficient of friction. The hard bearing phase of Al\(_2\)O\(_3\) and soft self-lubrication phase of PTFE in the coatings are likely to further improve their tribological performances. The aim was to adjust the particle contents in the composite coating and to obtain a comprehensive layer with low friction and good wear resistance. To explore the effect of particle contents on the tribological performance, friction tests were carried out.

Table 1
The main components of nickel electroplating solution.

<table>
<thead>
<tr>
<th>Compositions</th>
<th>Concentration (g·L(^{-1}))</th>
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</thead>
<tbody>
<tr>
<td>NiSO(_4)·6H(_2)O</td>
<td>200</td>
</tr>
<tr>
<td>NiCl(_2)·6H(_2)O</td>
<td>45</td>
</tr>
<tr>
<td>H(_3)BO(_3)</td>
<td>40</td>
</tr>
<tr>
<td>Lauryl sodium sulfate</td>
<td>0.5</td>
</tr>
<tr>
<td>1, 4-Butynediol</td>
<td>0.4</td>
</tr>
<tr>
<td>Saccharin sodium</td>
<td>1</td>
</tr>
</tbody>
</table>

Table 2
Composite coatings and preparation conditions.

<table>
<thead>
<tr>
<th>Sample no.</th>
<th>Electrophoretic parameters</th>
<th>Electrodeposited cathodic current density (A·dm(^{-2}))</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>PTFE concentration (g·L(^{-1}))</td>
<td>Al(_2)O(_3) concentration (g·L(^{-1}))</td>
</tr>
<tr>
<td>1</td>
<td>12</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td>12</td>
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<tr>
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<td>6</td>
</tr>
<tr>
<td>5</td>
<td>12</td>
<td>8</td>
</tr>
<tr>
<td>6</td>
<td>0</td>
<td>12</td>
</tr>
</tbody>
</table>

Fig. 4. Schematic diagram of experimental apparatus for electrodeposition.
Fig. 6 shows the friction curves of the pure Ni and composite coatings (No.1–6) with sliding time. The results show that the Ni coating has the highest friction coefficient, which is approximately 0.65. The Ni–nano-Al2O3 composite coating also has a relative high friction coefficient of about 0.58. Compared with the pure Ni coating, it can be found that the Al2O3 particles embedded in the coating show little anti-friction effect. The Ni–PTFE composite coating exhibits the lowest friction coefficient (0.11) among all the samples. The major reason should be the self-lubricating property of PTFE particles. As pointed by Seung and Andreas [21], the PTFE-based coatings showed excellent frictional characteristics and good wear resistance attributed to the beneficial effects of the generated wear debris at the interface. Sample No.2 is the Ni–nano-Al2O3–PTFE composite coating consisting of 24.17 wt.% PTFE particles and 8.11 wt.% nano-Al2O3 particles. Compared with the Ni–PTFE composite coating, the friction coefficient of the Ni–nano-Al2O3–PTFE composite coating (sample No.2) increases apparently. The result closely related to the decrease of PTFE particles and the increase of the nano-Al2O3 particles. The friction coefficients of samples No.3 and No.4 continue to increase in contrast with sample No.2, which is closely related to the increase of Al2O3 content in the composite coating. The existence of nano-Al2O3 particles leads to abrasive wear, which could be the main reason [22] This group of test indicates that the frictional property of the coatings could be adjusted by changing the particle concentrations in the coatings.

Fig. 7 shows the wear test results of the samples under different load conditions and Fig. 8 gives the corresponding morphologies of worn surfaces. As can be seen from Fig. 7, the Ni–nano-Al2O3 composite coating presents the best anti-wear performance. The width and depth of wear scar in the Ni–nano-Al2O3 composite coating (see Fig. 7a) are smaller than the others. As Shrestha [18] pointed out, the Ni–Al2O3 composite coating (sample No.2) shows the lowest friction coefficient (0.11) among all the samples. The major reason should be the self-lubricating property of PTFE particles. As pointed by Seung and Andreas [21], the PTFE-based coatings showed excellent frictional characteristics and good wear resistance attributed to the beneficial effects of the generated wear debris at the interface. Sample No.2 is the Ni–nano-Al2O3–PTFE composite coating consisting of 24.17 wt.% PTFE particles and 8.11 wt.% nano-Al2O3 particles. Compared with the Ni–PTFE composite coating, the friction coefficient of the Ni–nano-Al2O3–PTFE composite coating (sample No.2) increases apparently. The result closely related to the decrease of PTFE particles and the increase of the nano-Al2O3 particles. The friction coefficients of samples No.3 and No.4 continue to increase in contrast with sample No.2, which is closely related to the increase of Al2O3 content in the composite coating. The existence of nano-Al2O3 particles leads to abrasive wear, which could be the main reason [22] This group of test indicates that the frictional property of the coatings could be adjusted by changing the particle concentrations in the coatings.

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prepared by the two-step method had excellent tribological properties. First, the particles incorporated into the metal matrix made particles expose to the surface of the coatings. The existing manner of the particles may reduce the wear of the coating during the wear test. Second, there is a high content of the particles in the composite coatings, which can develop the high wear resistance of the coatings. Similar result was also reported by Hou et al.[10] using hard particles of SiC and the wear resistance of the layer increases with increasing content of SiC particles. The anti-wear performance of the Ni–PTFE composite coating is inferior to the Ni–nano-Al2O3 composite coating and the width and depth of the wear scar in Ni–PTFE coating under the load of 8.9 N are bigger than the lower load of 4.7 N and 6.6 N, which may be relevant to the property of the PTFE particle.

Sample No.2 has the worst wear resistance which is even worse than the Ni–PTFE composite coating (see Fig. 8). The distributed nano-Al2O3 fails to increase the wear resistance of the coating, but exacerbates the wear. As can be seen from Fig. 8b, a lot of grooves and scratches were observed on the worn surface, indicating severe wear. The result shows that low concentration Al2O3 particles can hardly prevent further wear. In the early stage of the wear, the Al2O3 particles can easily fall off from the substrate. Since the hardness of the Al2O3 particle is higher than Ni, the particles pressed into the coatings, acting as abrasive grain, will exacerbate the wear process. The wear resistance of the sample No.3 is worse than the Ni–PTFE composite coating and the wear mechanism is similar to sample No.2. The wear resistance of the sample No.4 is better than the Ni–PTFE composite coating. As can be seen from Fig. 8c, the uniformly distributed black lubricating film produced by PTFE and Al2O3 particles coexists in the worn surface. The massive Al2O3 particles, acting as bearing phase, hinder the further wear and

<table>
<thead>
<tr>
<th>Picture no.</th>
<th>Corresponding sample no.</th>
<th>Ni (wt.%)</th>
<th>PTFE (wt.%)</th>
<th>Al2O3 (wt.%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(a)</td>
<td>(1)</td>
<td>68.83</td>
<td>31.17</td>
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<tr>
<td>(b)</td>
<td>(2)</td>
<td>67.72</td>
<td>24.17</td>
<td>8.11</td>
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<td>(c)</td>
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<td>(d)</td>
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<tr>
<td>(e)</td>
<td>(6)</td>
<td>55.74</td>
<td>0</td>
<td>44.27</td>
</tr>
</tbody>
</table>

Table 3
Particle content in the composite coatings.

Fig. 5 (continued).

Fig. 6. The relation curves that friction varied with time under dry sliding conditions (the ratio in the picture meant the concentration ratio of nano-Al2O3 and PTFE in the electrophoretic liquid).
the black lubricating film alleviates the wear. However, compared with the Ni–nano-Al2O3 composite coating, the content of Al2O3 in sample No.4 is limited. Hence, it performed inferior wear resistance.

In general, for the excellent self-lubrication properties of PTFE, the Ni–PTFE coating shows the lowest friction coefficient. However, the load capacity of the coating is limited because of the low hardness of the PTFE and severe wear was found according to the appearance of wear surface. Comparison of the wear data between Ni–PTFE and Ni–nano-Al2O3 coatings shows that the co-deposition of the Al2O3 particle in Ni matrix has improved the wear resistance and the wear scar width decreased sharply. It seems that there is a direct relation between the hardness and wear resistance. For Ni–nano-Al2O3–PTFE coatings, the parallel tracks were found showing that the abrasion wear is the dominant wear mechanism. The poor bonding between the particles and the Ni could contribute pullout of the particles, which could intensify the wear destruction.

4. Conclusions

In this paper, Ni–nano-Al2O3–PTFE composite coatings have been successfully prepared by the electrophoretic and electro-deposition process. Compared with the conventional electroless composite plating, this method can significantly increase the particle content in the composite coating. Since the hard nano-Al2O3 particles can act as bearing phase and the soft PTFE particles as self-lubrication phase, the tribological performance of the composite coating was greatly improved. The main conclusions are as follows.

![Fig. 7. The wear resistance comparison of the samples under different load conditions.](image)

(a) Width of wear scar  
(b) Depth of wear scar

![Fig. 8. Morphology of worn surface under dry sliding conditions: (a) Ni–PTFE, (b) Ni–nano-Al2O3–PTFE (sample No.2), (c) Ni–nano-Al2O3–PTFE (sample No.4), and (d) Ni–nano-Al2O3.](image)
(1) Using the electrophoretic co-deposition method, a uniform electrophoresis sediment layer containing the nano-Al₂O₃ and PTFE particles could be formed on substrate. Moreover, the ratio of particle content could be adjusted by changing the particle concentration in the electrophoretic suspension.

(2) The electrophoretic and electro-deposition method could significantly increase the contents of nano-Al₂O₃ and PTFE particles in the composite coating. In this study, the content of PTFE and nano-Al₂O₃ particles in the composite coating could reach 31.17 wt.% and 44.27 wt.%, respectively.

(3) Compared with the pure Ni coating, the existence of PTFE in the composite coating could decrease friction. While the nano-Al₂O₃ in the layer, acting as bearing phase, could improve the anti-wear properties significantly.

(4) The Ni–nano-Al₂O₃–PTFE composite coating with both anti-friction and wear resistance can be prepared by optimizing the ratio of particle content in the coatings.

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